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Indian Standard
METHOD OF TEST FOR PULP

PART 19 DETERMINATION OF ALKALI SOLUBILITY OF PULP

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August 1985

Indian Standard

METHOD OF
TEST FOR PULP

PART 19 DETERMINATION OF ALKALI SOLUBILITY OF PULP

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Indian Standard

METHOD OF TEST FOR PULP

PART 19 DETERMINATION OF ALKALI SOLUBILITY OF PULP

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 January 1985, after the draft finalized by the Paper and Its Products Excluding Packaging Materials Sectional Committee had ??? approved by the Chemical Division Council.

0.2 The method is mainly intended for the investigation of bleached pulps, but may, however, also be used with unbleached pulps, for example, in the different stages of manufacture of bleached pulp.

0.3 The object of both the standards [this standard and IS : 6213 (Part 20)-1984* (under print)] is to permit the study the behavior pulps in the presence of alkali solution, but their fields of application different, while this standard describes the volumetric determination of the alkali-soluble constituents of the pulp and is applied preferably to the control of bleached pulps, IS : 6213 (Part 20)-1984* (under print) describes the gravimetric determination of the alkali-insoluble constitutents of the pulp and applies to all catagories of pulp.

0.4 This standard is based on ISO 692-1982 'Pulps — Determination of alkali solubility' issued by the International Organization for Standardization (ISO).

0.5 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard specifies a method for the determination of the solubility of pulp in cold sodium hydroxide solutions of various and fixed concentrations.

---

*Method of test for pulp: Part 20 Determination of alkali resistance of pulp (under print).

†Rules for rounding off numerical values (*revised*).

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 S-Value — Alkali solubility; the soluble fraction expressed as a percentage by mass of the oven-dry pulp.

2.2 S_{18}, S_{10} or S_c — S-values in which the indices 18, 10 or C refer to the chosen concentration, in grams of sodium hydroxide per 100 g of solution.

3. PRINCIPLE OF THE METHOD

3.1 Treatment of the pulp with sodium hydroxide solution and oxidation of the dissolved organic matter with potassium dichromate. Titration of the excess potassmm dichromate and calculation of the amount of cellulose equivalent to the potassium dichromate consumed.

4. QUALITY OF REAGENTS

4.1 Unless otherwise specified, pure chemicals and distilled water (see IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

5. REAGENTS

5.1 Sodium Hydroxide — solution of known concentration, containing less than 1 g of sodium carbonate per litre (see Note), for example:

a) —5.39 ± 0.03 mol/l solution, containing 18.0 ± 0.1 g of sodium hydroxide per 100 g of solution, equivalent to 215.5 ± 1.0 g of sodium hydroxide per litre.

b) -2'77 ± 0.03 mol/l solution, containing 10.0 ± 0.1 g of sodium hydroxide per 100 g of solution, equivalent to 110.9 ± 1.0 g of sodium hydroxide per litre.

NOTE — The sodium hydroxide solution may be conveniently prepared as follows:

Dissolve a quantity of solid sodium hydroxide in an equal mass of water and allow the supended sodium carbonate to settle. Decant the supernatant liquid and dilute with carbon dioxide-free water to the appropriate concentration. Check by titration with standard acid solution.

5.2 **Concentrated Sulphuric Acid** — Not less than 94 percent (*v/v).*

NOTE — If the concentration of the sulphuric acid is less than 94 percent (*v/v),* the temperature will not reach the 125 to 130°C required during the oxidation.

---

*Specification for water for general laboratory use (second *revision*).

5.3 Potassium Dichromate — About 0.067 mol/1 solution in 2.7 mol/1 sulphuric acid solution; 20 g of potassium dichromate ($K_2Cr_2O_7$) and 150 ml of sulphuric acid (Q_{20} = 1.84 g/ml) per litre of solution.

5.4 Ammonium Iron (II) Sulphate — Approximately 0.1 mol/1 solution [concentration known to an accuracy of ±0.000 2 mol/1): 40 to 41 g of ammonium iron (II) sulphate hexahydrate ($NH_4)_2SO_4$ $FeSO_4.6H_2O$] and 10 ml of sulphuric acid (Q_{20} = 1.84 g/ml) per litre of solution.

This solution is not stable and its concentration shall be checked every day.

NOTE — In order to restore the original concentration of the ammonium iron (II) sulphate solution, a reducingagent can be placed between the supply bottle and the burette. Prepare the reducing agent as follows:

Wash metallic cadmium (which passes a sieve of aperture size 1.4 to 2.0 mm) with water to remove fine particles. Treat the metal for about 5 min with a 2 percent solution of mercury (II) nitrate [$Hg(NO_3)_2.H_2O$] or mercury (II) chloride ($HgCl_2$) containing 5 ml of concentrated nitric acid per litre, and then wash the amalgamated metal.

The ammonium iron (II) sulphate solution may be standardized against potassium dichromate as a primary standard.

The concentration of the ammonium iron (II) sulphate solution will stay constant if 5 g of aluminium shavings, of purity greater than 99.99 percent, are added to 10 litres of solution.

5.5 Phosphoric Acid — 85 percent (*v/v*) (Q_{20} = 1.70).

5.6 Ferroin Indicator Solution— 15 g of 1,10 phenanthroline monohydrate ($C_{12}H_8N_2.H_2O$), or 16 g of 1,10 phenanthroline hydrochloride ($C_{12}H_8N_2.HCl.H_2O$), and 7 g of iron (II) sulphate ($FeSO_4.7H_2O$) per litre of solution, or

5.7 Sodium Diphenylaminosulphonate Indicator solution — Dissolve 0.1 g of sodium diphenylaminosulphonate ($C_{12}H_{10}NSO_3Na$) in water and dilute to 100 ml.

6. APPARATUS

6.1 Stirring Equipment — With a propeller-type agitator made of stainless steel or other corrosion-resistant material. The angle of the blades shall be adjusted so that air is not introduced into the pulp suspension during stirring.

NOTE — Suitable equipment is shown in Fig. 1. A statisfactory motorlis one of 15 W, operating at a rotational frequency between about 28 and 24s^{-1}

6.2 Constant Temperature Bath — Capable of maintaining a temperature of 20 ± 0.2°C.

6.3 Filter Crucibles or Funnels — Of capacity 50 ml, made of alkali-resistant material, with a sintered glass disc of pore size greater than 16 μm but less than or equal to 40 μm.

6.4 **Suction Flasks** — For the crucibles or funnels.

6.5 **Balance** — Accurate to 1 mg.

6.6 **Reaction Vessel** — Of capacity 200 ml and tall in shape.

7. PREPARATION OF THE TEST SAMPLE

7.1 If the pulp is in sheet form, tear into pieces of size approximately 5 × 5 mm. If it is in slush form, remove the water by suction, press between blotters and dry at a maximum temperature of 60°C. Before weighing, condition the sample for not less than 20 min in the atmosphere near the balance.

8. PROCEDURE

8.1 Test Portion — Weigh, to the nearest 0.005 g the equivalent amount of approximately 1.5 g of oven-dry pulp. Then immediately weigh two test portions for the determination of the dry matter content in accordance with IS : 6213 (Part 21)-1984*.

8.2 **Determination** — By means of a pipette, transfer 100.0 ± 0.2 ml of the chosen sodium hydroxide solution to the reaction vessel. Adjust the temperature to 20 ± 0.2°C (*see* Note 1) by placing it in the constant temperature bath.

NOTE 1 - The solubility in 18 percent (m/m) sodium hydroxide solution is not affected by variations of a few degrees in temperature. At this concentration, the temperature may be kept at 20 ± 2°C.

The solubility in sodium hydroxide solution of lower concentration (for example, 10 percent (*m/m*) is much more dependent on temperature. At this lower concentration, the temperature should be kept at 20 ± 0.2 *"C.*

8.3 Add the test portion to the sodium hydroxide solution and allow to swell for 2 min. Stir in the reaction vessel for 3 min or until the pulp is completely disintegrated (see Note 2). Lift the stirrer from the reaction vessel. Some fibres and sodium hydroxide solution may remain on the stirrer when, it is removed, but at the low concentration used, this small loss may be neglected. Maintain the reaction mixture at 20 ± 0.2°C for a period of 60 min from the time the pulp was brought into contact with the sodium hydroxide solution.

NOTE 2 — An increase in stirring time does not significantly affect the alkali solubility, but too low a value will be obtained if disintegration is not complete. Keep stirring, therefore, until the test portion is completely disintegrated.

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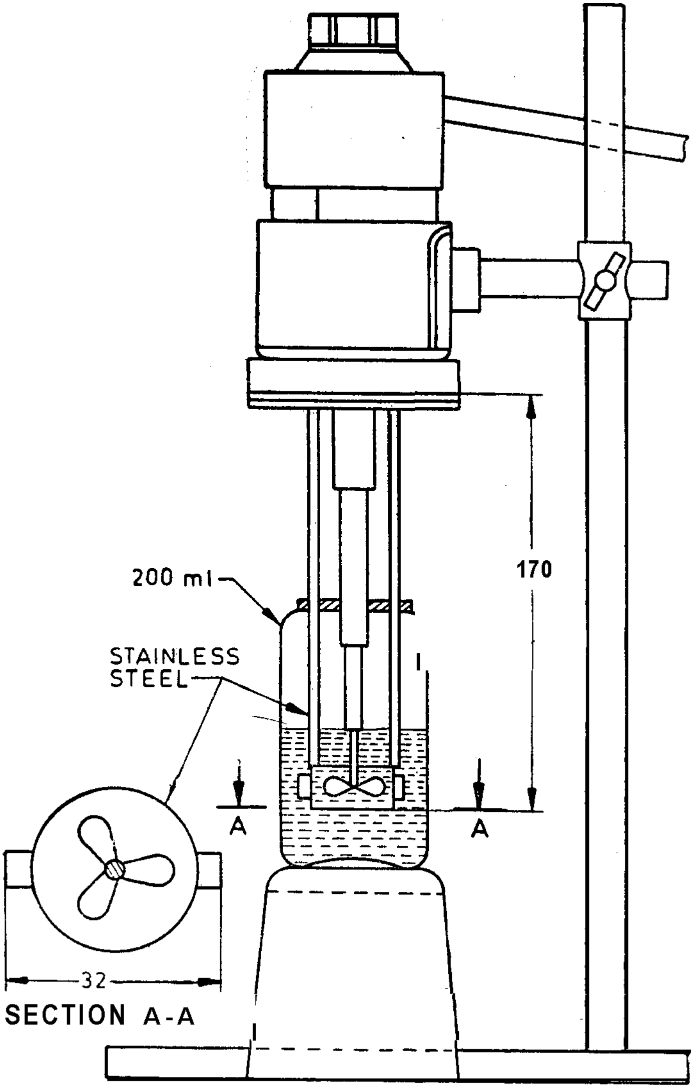
*Methods of test for pulp: Part 21 Determination of dry matter content.



All dimensions in millimetres.

FIG. 1 PULP DISINTEGRATOR

8.4 At the end ??? 60 min, stir the slurry with a glass rod and filter with slight suction through the filter crucible or funnel avoiding passage ??? air through the residue. Reject the first 10 to 20 ml and collect the next 40 to 50 ml of the filtrate in a clean bottle or flask.

8.5 By means of a pipette, transfer 10.0 ml (*see* Note 3) of this filtrate to a 250 ml conical flask. Add, by means of a pipette, 10.0 ml ??? the potassium dichromate solution and then carefully add, with swirling, 30 ml of the concentrated sulphuric acid. Check that the temperature is between 125 and 130°C

NOTE 3 — When normal dissolving pulps are tested, 10 ml ??? filtrate is a suitable aliquot. If the alkali solubility it greater than 16 percent (*m/m*), reduce the volume the aliquot portion to 5 ml ??? the amount of sulphuric acid to 25 ml. If the alkali solubility is less than 5 percent (*m/m*), use 20 ml ??? filtrate and 45 ml of the sulphuric acid.

In the blank test, use the corresponding volumes of sodium hydroxide solution ??? sulphuric acid.

8.6 Allow the hot solution to remain at a temperature ??? 120°C for 10 mm to complete the oxidation. Cool the flask to room temperature.

8.7 According to the case, proceed as follows:

a) To the cold solution, add 50 ml ??? water. Cool again add 2 drops ??? ferroin indicator solution and titrate with the freshly standardized (*see* Note under **5.4**) ammonium iron (II) sulphate solution to a violet colour.

b) Dilute the cold solution with water to a volume ??? about 100 ml and add 5 ml the phosphoric acid. Cool again, ??? titrate rapidly with the freshly standardized ammonium iron (II) sulphate solution until about 90 percent of the required amount added. Then, by means ??? a pipette, add 1 ml of the sodium diphenylaminosulphonate indicator solution, and titrate further without delay to the colour change from dark brown through violet to bright green (*see* Note 4).

NOTE 4 — In excess acid, potassium dichromate solution, the indicator is partly oxidized, which not only results in potassium dichromate consumption, but also in other than original colour characteristics. As the oxidation depends on such factors as relative amounts and concentrations of potassium dichromate and indicator, the excess potassium dichromate must be reduced as quickly as possible. This is done most effectively by adding the indicator no sooner than after neutralization of about 90 percent of the excess potassium dichromate. When the titration is completed with out delay, the indicator error has been found to be negligible.

8.8 Carry out a blank test, substituting 10 ml of the chosen sodium hydroxide solution for the filtrate and using approximately the same temperature and time to complete the titration (*see* Note 5).

NOTE 5 — Alternatively, an iodometric titration procedure can be used, but its use shall be stated in the test report.

Transfer the cold solution, after oxidation, to a 1 000 ml conical flask, using 500 ml of water. Add 2 g of potassium iodide (KI), maintaining the temperature below 10°C, swirl to dissolve and mix; then allow to stand for 5 min. Titrate with standardized 0.1 mol/l sodium thiosulphite solution ($Na_2S_2O_3$), adding powdered starch indicator when the yellow colour of the iodine has nearly disappeared. The end point is indicated by a change from deep blue to light green. Carry out a blank test, substituting a 10 ml portion of the sodium hydroxide solution for the filtrate. Calculate the result as described in 9 . 1 , substituting for V_1 and V_2 the corresponding volumes of sodium thiosulphate solution and for C the concentration of the solution.

8.9 Carry out two determinations on each sample.

9. EXPRESSION OF RESULTS

9.1 Method of Calculation and Formula — The alkali solubility, Sc, expressed as a percentage by mass, is given by the formula:

$$\frac{6{\cdot}85\,(V_2 - V_1) \times c \times 100 \times 100}{1\,000 \times m \times V} = \frac{68{\cdot}5\,(V_2 - V_1) \times c}{m \times V}$$

where

V = the volume, in millilitres, of filtrate used in the oxidation;

V_1 = the volume in millilitres, of ammonium iron (II) sulphate solution consumed in the titration of the test portion;

V_2 = the volume, in millilitres, of ammonium iron (II) sulphate solution consumed in the blank test;

c = the concentration, in moles per litre, of the ammonium iron (II) sulphate solution;

m = the mass, in grams, of the test portion, calculated on an oven-dry basis;

6.85 the empirical factor, in milligrams, indicating the amount of cellulose equivalent to 1/6 m mol of potassium dichromate.

9.1.1 A sample calculation is given in Appendix A.

NOTE — Theoretically, 1/6 m mol of potassium dichromate corresponds to 6.75 mg of cellulose or other hexosans, and 6.60 mg of pentosans. In general, the soluble components of pulp consume less oxidizing agent than the theoretical amount because they contain oxycellulose. Therefore, a somewhat higher figure 6.85 mg, is used in this method.

9.2 **Precision and Expression** of **Results** — The results of two determinations should agree to within 0.3 percent.

Report the mean alkali solubility to one decimal place; using the symbols S_{10}, S_{18}, etc.

NOTE— For pulps containing less than 0.1 percent ash and other non-carbohydrate materials, the value ($100 - S_c$) approaches the value R_c determined by the method for alkali resistance of pulps.

10. TEST REPORT

10.1 The test report shall include the following particulars:

a) A reference to this standard;
b) The results and the method of expression used;
c) Any unusual features noted during the determination; and
d) Any operation not specified in this standard, or regarded as optional.

APPENDIX A

(*Clause* 9.1.1)

EXAMPLE OF CALCULATION

Mass of the air-dry test portion	1.735 g
Dry matter content determined on a separate test portion	92.4 percent
Mass the test portion calculated the oven-dry basis (m)	1.604 g
Volume ammonium iron II sulphate solution consumed in the blank test (V_2)	41.1 ml
Volume ammonium iron II sulphate solution consumed in the titration of the test portion (V_1)	15.5 ml
Concentration of the ammonium iron (II) sulphate solution (c)	0.100 5 mol/1
Volume of filtrate used in the oxidation (V)	10 ml

Solubility in sodium hydroxide solution (S_c) percent by mass:

$$= \frac{68{\cdot}5\,(\,41{\cdot}1 - 15{\cdot}5\,)\,0{\cdot}100\,5}{1{\cdot}604 \times 10}$$

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